Temperature and Temperature Gradient Measurement Method in the Laser-Heated Diamond Anvil Cell

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Abstract

The determination of material properties under the conditions of the Earth's interior requires experiments performed at temperatures up to 6000 K, and pressures in excess of 100 million times atmospheric pressure. To achieve these conditions, samples of less than 100 ng are heated with an infrared laser focused to an area of about $7x10^{-6}cm^2$ inside a laser heated diamond anvil cell (LHDAC). The temperature measurement method is based on spectroradiometry of blackbody thermal emission. Most current methods suffer from chromatic aberrations in which blue light is focused more tightly than red light due to wavelength-dependent refraction through optical components. Existing spectroradiometry systems are subject to aberrations from the dispersion of the diamond and refractive optics. These systems also do not constrain temperature and wavelength-dependent emissivity. Although the peak temperature measurement is accurate, the temperature gradient can be underestimated by as much as 100% (Kavner and Panero, 2004). I present a calibration of a newly designed optical system to accurately measure the temperature and temperature gradient of a high pressure sample. The redesigned optical system splits the emitted, focused light from a small sample into two paths. One system, at the end of the first path is a spectrometer and CCD that collects intensity at the center of the sample where the light is selected by a 10 μm slit. The other path leads to a high-dynamic range CCD camera that collects a 15x magnified image of the sample to measure total intensity as a function of position. With this data, the peak temperature, least affected by chromatic aberrations, provides a scaling factor for the spatial measurement to produce a two dimensional measure of the temperature across the sample. Data sets are collected simultaneously on

both optical paths and then processed in a manner to relate temperature to intensity on an image of the sample. In this way, temperature can be measured at any point on the sample *in situ*. By use of a scaling factor in temperature measurements this setup lessens the effects of chromatic aberrations as well as creates opportunity to perform experiments which necessitate a higher degree of accuracy than allowed by current methods.

I. Introduction

Due to the wide range of temperatures and pressures in the earth, minerals exhibit drastic changes in properties with change in depth. In order to understand the behavior of deep earth materials it is necessary to perform experiments under similar conditions. Many of these experiments involve the determination of temperature inside the laser-heated diamond anvil cell. As experiments become more advanced and sample size decreases in many modern experiments, the uncertainty in temperature measurement due to chromatic aberrations and temperature gradients becomes increasingly significant.

In this paper a discussion of the design, calibration, and testing of an optical system to measure the temperature and temperature gradient in the LHDAC will be presented based on methods described in Kavner and Panero (2004). Four methods are described in their work, and are integrated into the system and data analysis process. By using these methods in conjunction it is possible to mitigate some of the common problems in temperature determination and place more accurate limits on the behavior of minerals in the deep earth.

A planned application for this system is to perform iron melting experiments in the LHDAC. The goal is to reduce the level of uncertainty in current phase boundaries by taking more accurate measurements of melting as related to temperature and pressure. A

checkerboard pattern of iron 56/57 will be placed in the LHDAC. Melting will be determined by a level of mixing separable from simple diffusion by detecting the migration of iron isotopes. By relating the location of significant migration to the temperature of the location and the pressure in the cell, it will be possible to obtain many data points from a single heating episode. The Planck radiation function and the Stephan Boltzmann law are the basis for the temperature determination.

A. Planck Function

The Planck radiation function relates intensity to wavelength and temperature.

$$I(\lambda,T) = \frac{\varepsilon 2hc^2}{\lambda^5} \frac{1}{e^{\frac{hc}{\lambda kT}} - 1}$$

Where h is Planck's constant, λ is wavelength, k is Boltzmann's constant and c is the speed of light. ε, emissivity, is the ratio of grey-body intensity to ideal blackbody intensity. In this three variable equation, if it is possible to constrain two variables then the third is automatically defined. A simple explanation of blackbody radiation is that when a blackbody is at a certain temperature it emits a continuous spectrum where the spectral intensity at a certain wavelength for a given temperature is defined by the Planck radiation function.

B. Stephan Boltzmann Law

The Stephan Boltzmann law concerns the area under the Planck curves.

$$\int_0^\infty I(\lambda,T)d\lambda = \varepsilon\sigma T^4$$

This is the total, i.e., integrated intensity over all wavelengths. This area varies as a square of the temperature. The constants are the same as for the Planck function. Emissivity can be constant, dependent on temperature, wavelength, or be dependent on both variables. Emissivity can be viewed as a cause for deviation from ideal blackbody curves according to the Planck radiation function.

II. Common Problems

A. Chromatic Aberrations

One of the major problems facing nearly all of optical spectroradiometry is chromatic dispersion. Walter and Koga 2004 show that high peak temperatures, high spatial resolution, and large radial displacement (due to chromatic dispersion), create opportunity for miscalculations of temperature in modern high precision experiments in the LHDAC. The problem of chromatic dispersion is hard to avoid since the refractive index is wavelength dependent. Thus the use of any lens or diamond is guaranteed to introduce a bit of dispersion. These errors can be on the order of hundreds to thousands of degrees Kelvin (Walter and Koga, 2004).

The effect of chromatic dispersion is to have some wavelength of light not focused at a corresponding ideal area on the detector. It appears as if the wavelength is missing from where it would ideally be transmitted. Light from other regions on the object plane is displaced onto a given region of the image plane. In a sample where the temperature was uniform, chromatic dispersion would have no net effect since the blackbody emission from each point would be the same. Some optical systems claim that in the 500-900 nm range chromatic dispersion from 80 mm focal length lenses causes up to 200 micrometers of displacement. Similarly, the chromatic dispersion by a 2mm thick diamond would be

expected to be in the range of 2 μm for comparison. (Walter and Koga, 2004).

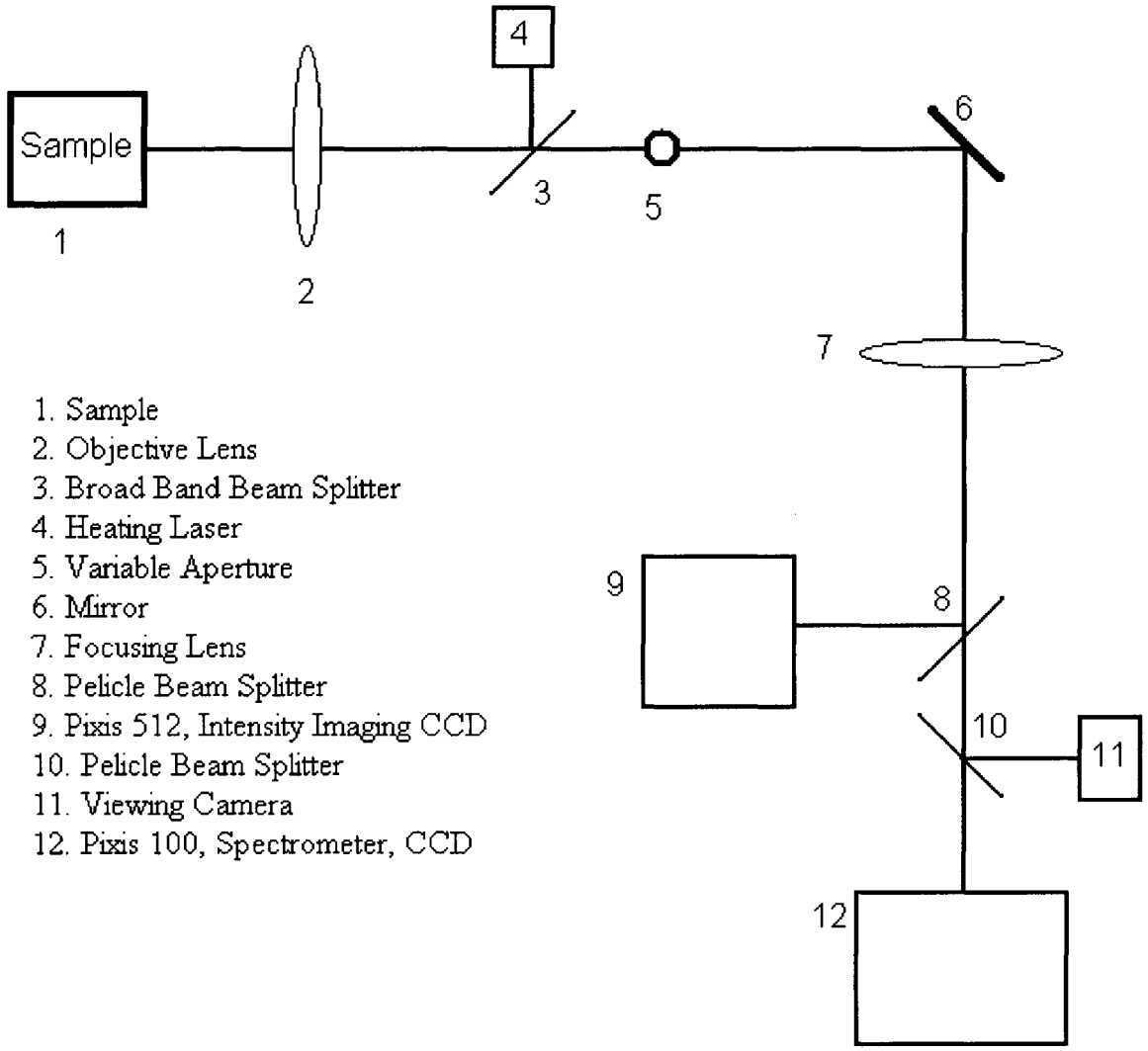
It is possible to minimize these effects in LHDAC experiments through several methods. Walter and Koga 2004 describe a method to create a scaling factor, our "system response" which is based on the use of a known intensity standard and data correction. The use of an adjustable aperture works to decrease the number of light rays from refracted areas on the object plane, thus allowing a greater percentage of paraxial rays from the sample to hit the detector. (Walter and Koga, 2004).

B. Temperature Gradients

The temperature is not constant across a sample in the LHDAC. In order to perform high accuracy experiments such as the ones necessary for phase boundary location, it is essential to account for the variation of temperature with position. According to Panero and Jeanloz (2001A,B) temperature gradients in the LHDAC arise primarily from spatially varying laser intensity and heat flow due to the high thermal conductivity of diamond. The temperature dependence of the sample thermal conductivity and the ratio of sample thickness to laser spot diameter help to define the temperature distribution (Panero and Jeanloz, 2001B). The temperature gradient will be especially important in the iron melting experiments described in the introduction since many samples will be arranged in the LHDAC during one heating episode.

III. Setup

Figure 1: A schematic of the optical table.



1. Sample
2. Objective Lens
3. Broad Band Beam Splitter
4. Heating Laser
5. Variable Aperture
6. Mirror
7. Focusing Lens
8. Pelicle Beam Splitter
9. Pixis 512, Intensity Imaging CCD
10. Pelicle Beam Splitter
11. Viewing Camera
12. Pixis 100, Spectrometer, CCD

The lab setup is based on the methods described by *Kavner and Panero 2004.* Blackbody radiation is emitted by the sample when it is heated by the laser. The emitted light passes through the objective lens (2) and through the variable aperture (4) before it is reflected by the mirror (6) through the focusing lens (7). After passing through the focusing

lens the light is split at the pellicle beam splitter (8) into two paths. One path leads to the Pixis 512 CCD which collects a relative intensity image of the sample. The second is a branched path leading to the viewing camera (11) and the Pixis 100 CCD and spectrometer (12). The viewing camera allows for sample image to be viewed during the focusing process. The spectrometer and Pixis 100 CCD is used to obtain intensity versus wavelength. In our experiment the objective and focusing lenses are Leitz μm/10, and 100 mm focal length BK-7 glass, respectively. The NIST-traceable light intensity standard lab protocol is described in detail on attached page A.

IV. Data Calibration

A. Spectrometer

Light from the sample undergoes narrow slit diffraction where the slit width is on the order of 10μm. The diffracted light is collected on a CCD (Fig.2). Wavelength calibration is preformed using a neon gas spectrum. A spectrum of the quartz-tungsten lamp at 6.5 Amps, 2886 K, is taken and corrected for the wavelength calibration (Fig. 3).

Figure 2: 6.5 amp spectrum of the quartz-tungsten lamp at 2886 K.

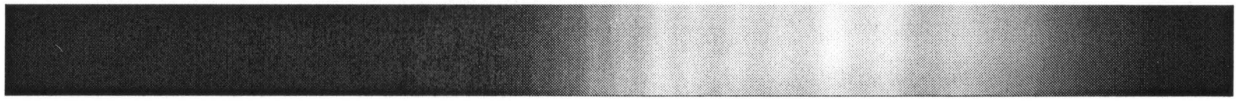
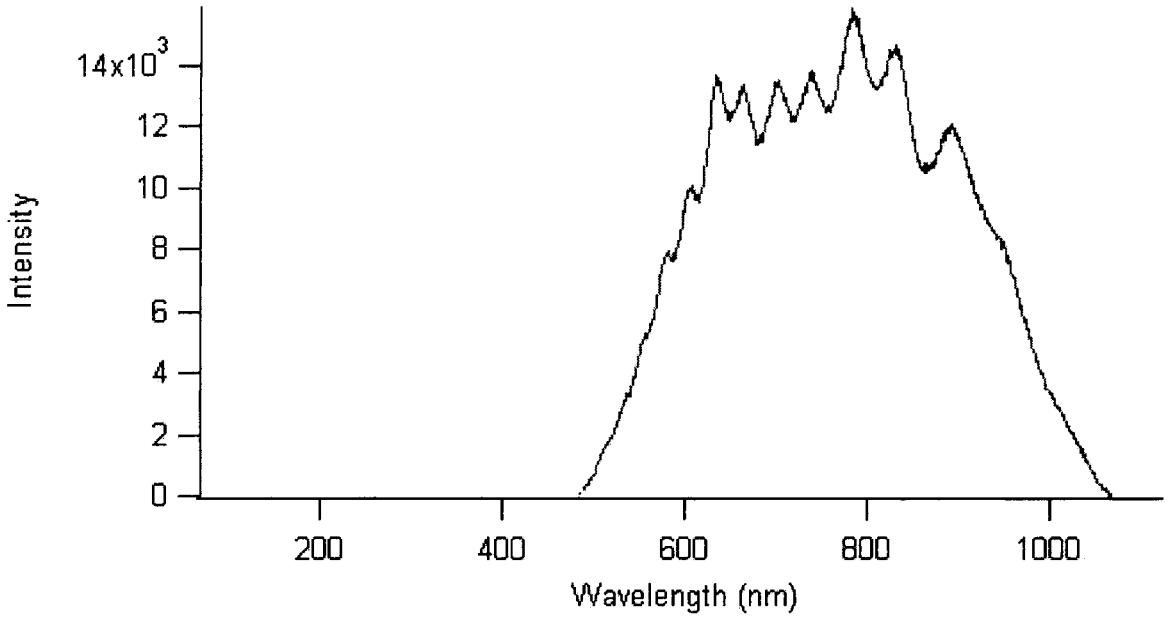


Figure 3: A profile across the spectrum in Fig. 2, corrected for wavelength position.



Using these measurements, a comparison of the expected Planck blackbody radiation function at 2886 K is used to create a scaling factor for future measurements taken on the spectrometer limb (Fig. 4). This scaling factor is intended to compensate for variations from ideal Planck blackbody radiative emissions caused by constant emissivity and dispersion by optical components. A proper description of this constant is "system response," since it is dependent on optical properties of components included in the system and represents the deviation from ideal behavior in response to the setup. Specifically, the ratio of measured data to expected values calculated by the Planck radiation function allows that the product of a sample's measured wavelength intensity and the inverse of the system response is the sample's relative intensity emitted at that wavelength (Fig. 5).

Figure 4: The system response is a scaling factor at each wavelength.

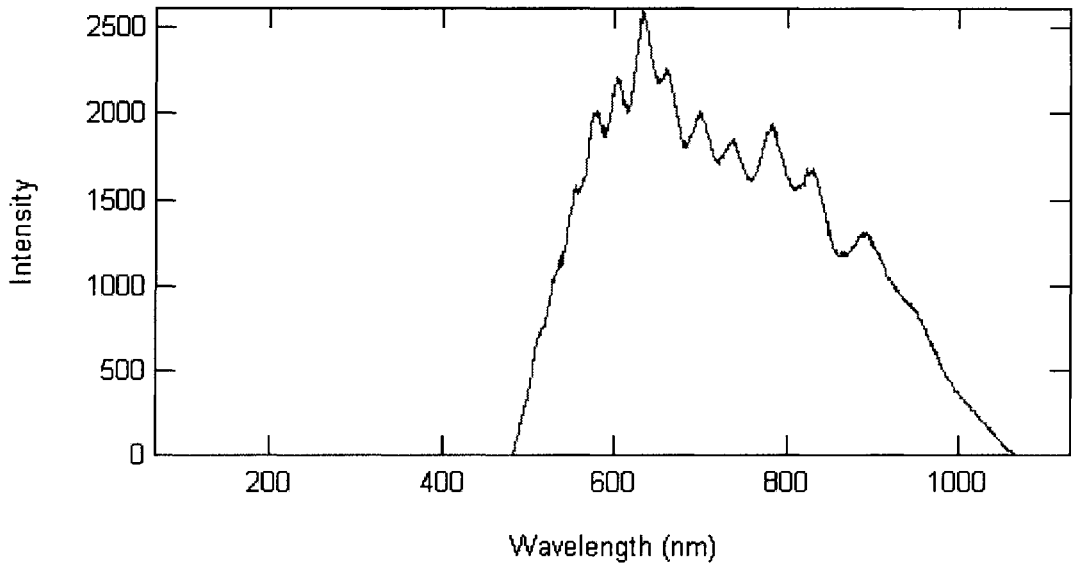
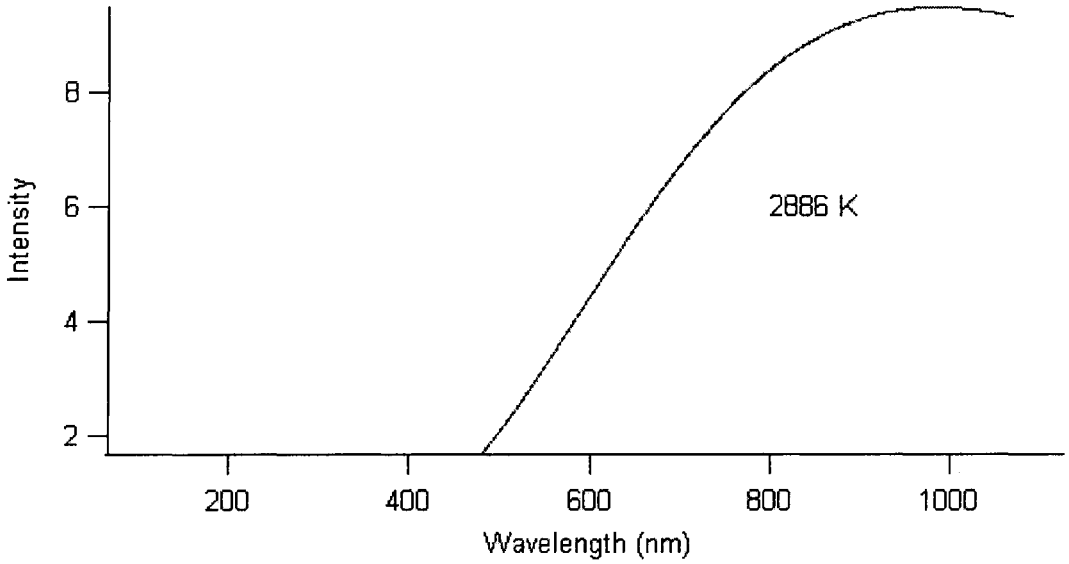


Figure 5: The 6.5 amp spectrum after correction using the system response.



To test the system, several unknown temperatures are calculated. Using a steady current power source, it is possible to create unknown temperature samples. Measured spectra (Fig. 6) can be corrected for the system response and the emitted wavelength intensities are fitted to the Planck radiation function to produce a peak temperature

measurement (Fig. 7).The results are used to define temperature as a function of current (Fig. 8). This final relation is used in the calibration of the high-dynamic range CCD camera.

Figure 6: Profiles of spectra at decreasing current.

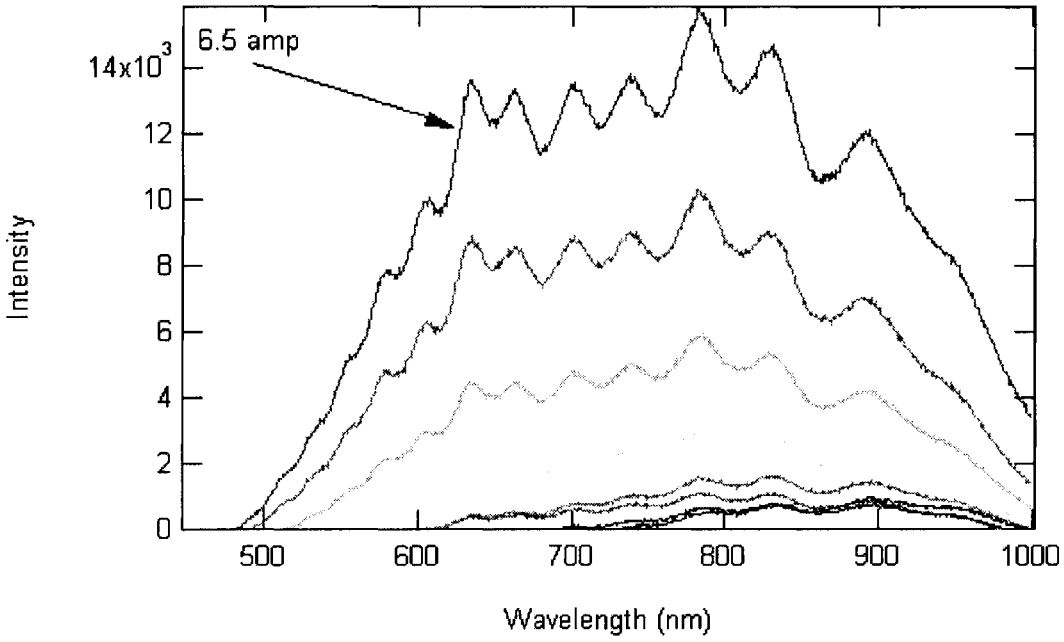


Figure 7: Spectra from Fig. 6 after correction for the system response, fitted to Planck curves.

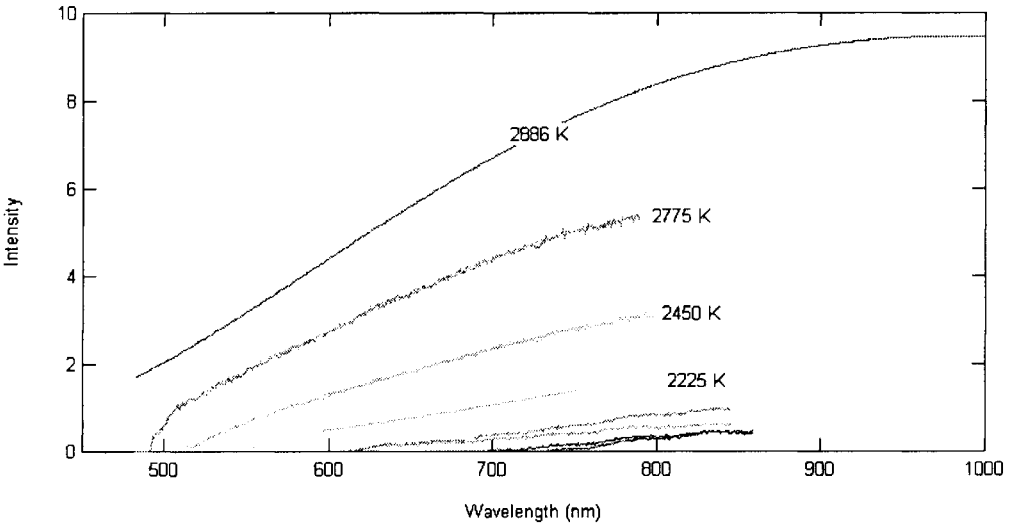
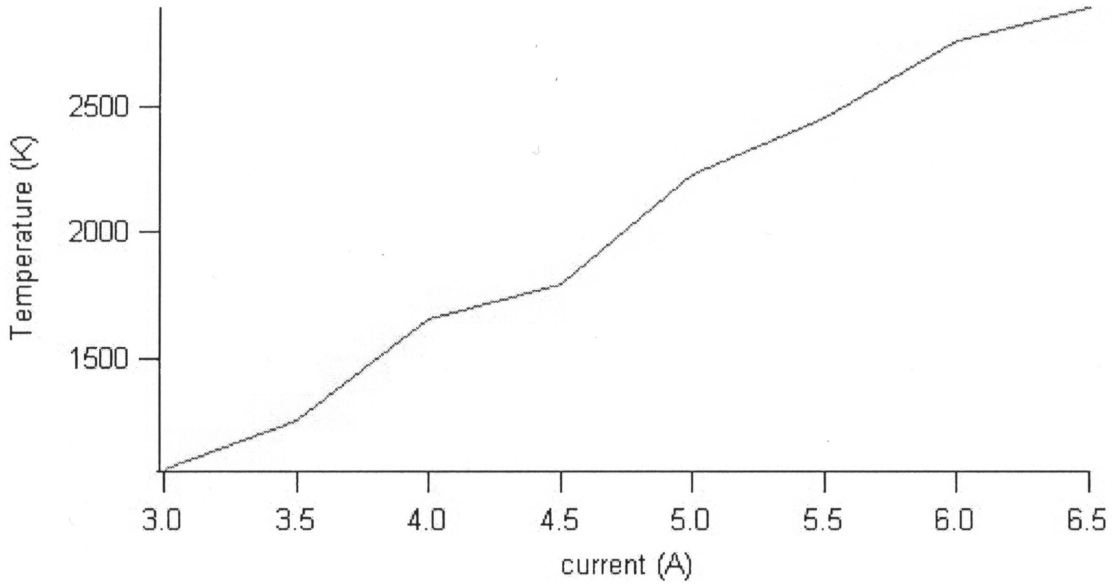


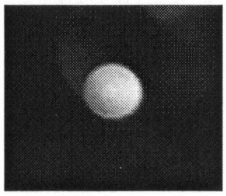
Figure 8: Temperature as a function of current.



B. Intensity Image

The second limb of the system is designed to collect a relative intensity image of the sample area (Fig. 9). The data set is in terms of relative total intensity versus position on surface of the sample area. In use of the lamp the center intensity is taken to be the measured intensity of blackbody emission for the peak temperature (Kavner and Panero 2004).

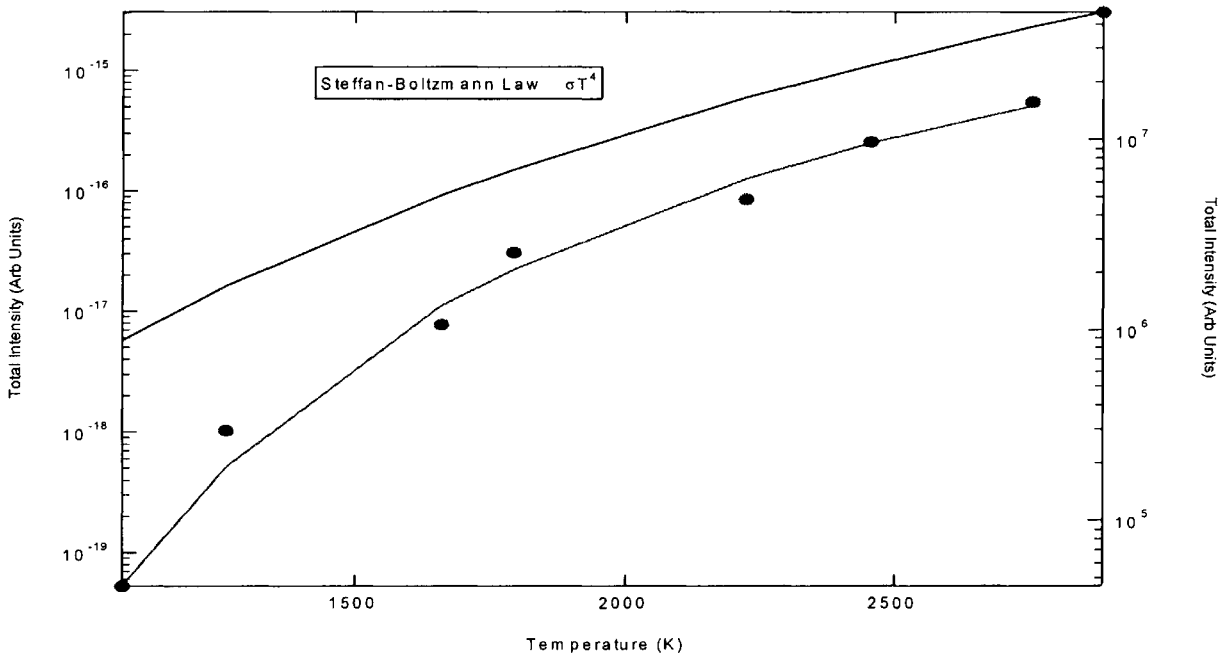
Figure 9: Relative intensity image of the quartz-tungsten lamp through a pinhole.



Next a function is created relating total measured relative intensity to temperature. This can be taken as a test of temperature dependent emissivity by comparing the measured

intensities for the sample temperatures to the system response-adjusted Stephan Boltzmann curve. Since the measurements (dots) made a good fit to their expected values it is taken that the effects of temperature dependent emissivity are minimal (Fig. 10).

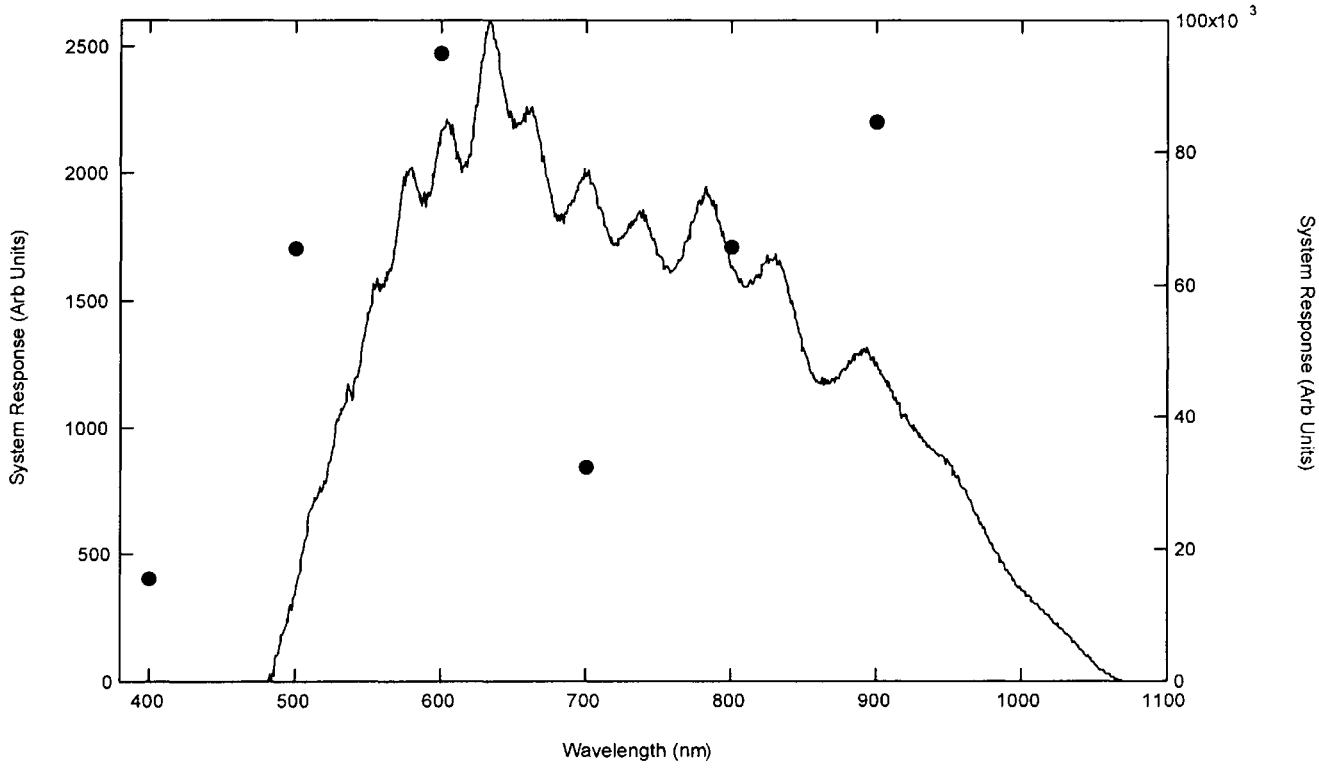
Figure 10: Stephen Boltzmann Law, top, shown against expected curve from system (line) and lab measured values (points)



In addition to collection of total intensity images it is possible to select the wavelength through the use of a notch filter. At each current, temperature, the sample is filtered to select wavelengths 900nm through 400nm in 100nm increments (Fig. 11). The ratio of measured relative intensity to expected relative intensity at each wavelength is taken as the system response in a similar method to the spectrometer system response. In this case the number of data points is limited by the number of filters on the notch filter wheel. The system response at each wavelength on the intensity image is compared to the system response on the spectrometer (solid line). The anomalous system response (dots) on the intensity image at the 700nm and 900nm wavelengths is due to low response of the

Pixis 512 camera itself near 700nm due to the coating of the front illuminated CCD chip (Fig. 11).

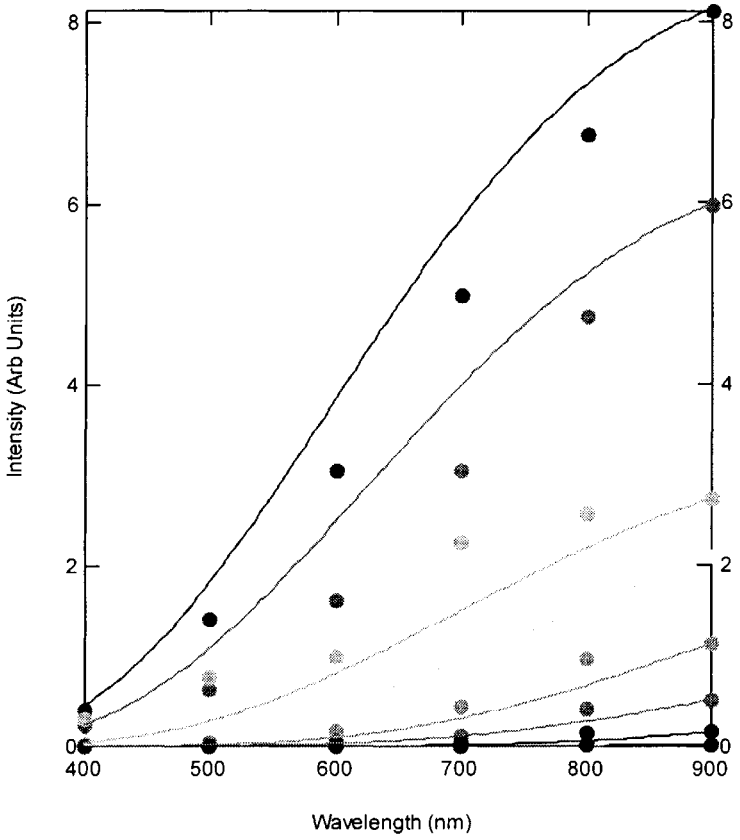
Figure 11: System response using spectrometer (solid) compared to system response at filtered wavelengths (points).



Temperature curves are constructed using the intensity image system responses at each wavelength and the measured intensity values (Fig. 12). These curves (as points) are then compared to the Planck curves for the expected temperatures (solid lines). The Planck curves obtained through the use of intensity imaging camera in conjunction with the notch filter have a different profile than the expected curves from the spectrometer data. This is

interpreted to be an effect of wavelength-dependent emissivity of the hot tungsten wire.

Figure 12: Curves from total intsnsity (solid) compared to measurements made using notch filter (points).



If the wavelength-dependent emissivity is constrained then use of the notch filter wheel could be another method to measure temperature, and would be well suited to applications where temperature-dependent emissivity is a factor. Temperature must be constant with respect to time in such applications, else the time to change filters and take an image of the sample would allow for a confounding temperature change.

V. Summary

Using the integrated intensity measured on the calibrated system, it is now possible to extract the temperature at any given point on a sample within the LHDAC. This method

helps to minimize the effects of temperature gradients and chromatic dispersion. Using intensity versus wavelength to calculate the temperature can be an indication of temperature and wavelength dependent emissivity. Measurement on other wire lamps (e.g. Mo, Fe, Ni, Pt) will help demonstrate the utility of this combined method for measuring emissivities. Other future plans are to use this optical setup and calibration technique to perform the iron 56/57 melting experiment described in the introduction.

VI. References

Kavner, A., and W. R. Panero, Temperature gradients and evaluation of thermoelastic properties in the synchrotron-based laser-heated diamond cell, Phys. Earth. Planet. Inter., 143-144, 527-539, 2004.

Panero, W. R., R. Jeanloz, Temperature gradients in the laser-heated diamond anvil cell, Journal of Geophysical Research, 106, 6493-6498, 2001A.

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Walter, M.J., K Koga, The effects of chromatic dispersion on temperature measurement in the laser-heated diamond anvil cell, Phys. Earth. Planet. Inter., 143-144, 541-558, 2004.

Page A: 45W Quartz-Tungsten Lamp Lab Protocol

Mounting the Lamp

When handling the lamp use chemwipes to avoid getting the lamp oily or dirty. Using open air lamp mount, install the calibrated lamp with the nipple facing away from the sensors and the positive end of the lamp in the positive end of the mount. Place the washers over the holes and then tighten the screws.

Getting the Power Supply Ready

Plug the appropriate end of the AC cord into the back of the power supply and the other end into an outlet. Turn the power supply on and hold "setup" button, select "set mode." Now select current mode and hit "set/enter." To set the current simply hold the "set/enter" button then use the arrows to adjust the amperage. The calibrated data is at 6.5 amps, and going above this value could damage the lamp.

Turning on the Lamp

Wear UV protective goggles when the lamp is on. Connect the power cable from the lamp to the power supply. Before you turn on the lamp double check the settings by hitting "display/select." In order to turn on the lamp simply hit the "lamp start" button. The lamp will take a short time to get to full power, check the current on the display before continuing.

Turning off the Lamp

To turn the lamp off simply push the lamp off button. The lamp will power down; watch the current on the power supply's display. Caution the lamp is hot.